Quantitative Analysis of Submicrometer Particles in the Scanning Electron Microscope (SEM) Utilizing the ζ Factor Approach

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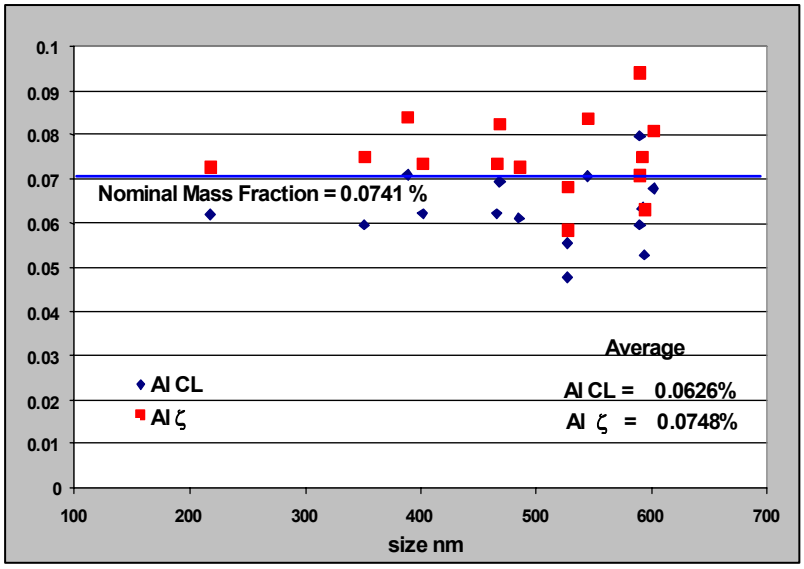
The analytical electron microscopy (AEM) approach for the quantitative analysis of ultrafine particles, 0.1 nm to 500 nm in size, involves the application of an elemental ratio procedure known as Cliff-Lorimer (CL). This procedure was designed for the AEM analysis of "infinitely" thin samples, for which there is negligible x-ray absorption. The basis for the CL approach is given in the equation to the right: Where k_{ab} is a proportionality constant calculated, using materials of known composition, for a given instrument at a given voltage, C_a and C_b are the concentrations of elements a and b, and I_a and I_b are the measured x-ray intensities.

$$C_a/C_b = k_{ab} * I_a/I_b$$

This investigation was undertaken to determine if the CL approach is applicable to the analysis of ultrafine particles in the SEM at 25 kV to 30 kV for which x-ray absorption is not negligible. In this study, a modified CL analysis procedure was employed that includes a correction for x-ray absorption known as the ζ factor; this factor was defined in *Ultramicroscopy* in 1996.

During the past year we have made a series of measurements on glass shards of known composition that had been ground to produce a large number of ultrafine particles. The particles were analyzed at 25 keV.



Plot of the Al concentration in ultrafine glass particles analyzed by both the CL method and the modified CL, which includes the ζ factor.

The results are shown in the figure, which plots the Al concentration analyzed by both the CL method and the modified CL that includes the ζ factor. The Al concentration from the CL method at a mass fraction of 0.063% has a negative bias compared to the nominal value of 0.074% as a result of not correcting for the absorption of the Al x-rays in the particles. In comparison, after the incorporation of the ζ factor into the CL method the mass fraction of Al percent is 0.075%, effectively removing the observed bias and bringing the average concentration more in line with the nominal value.

This study represents one of two methods we are currently investigating to improving the quantitative analysis of the ultrafine particles. The other approach involves reducing the accelerating voltage of the scanning electron microscope (SEM) to 5 kV or less for analysis. The rational for this approach is that the x-ray generation and emission volumes at the low voltage will be contained within the particle volume to a much greater degree than excitation at a higher but more conventional voltage such as 15 kV. Future plans involve the comparison of these two methods and their effectiveness for the quantitative analysis of ultrafine particles.

Traditionally ultrafine particles have been analyzed in analytical electron microscopes, which often require large capital investments in excess of $1M and have difficult sample preparation procedures. The SEM method described here could potentially offer a more economical approach.